1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 9

MUNICIPAL WASTE SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

G. C. RONAN, DIRECTOR
Laboratory Services Branch
Ministry of the Environment

1986

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT

SECTION 9

MUNICIPAL WASTE SAMPLES

INORGANIC TRACE CONTAMINANTS SECTION

D G STURGIS
and J C HIPFNER (editors)

Inorganic Trace Contaminants Section
Laboratory Services Branch
Ministry of the Environment

ALRR

ANNUAL QUALITY ASSURANCE PERFORMANCE REPORT 1986

INORGANIC TRACE CONTAMINANTS SECTION

SUMMARY

I. Introduction

The Inorganic Trace Contaminants Section of the Ministry of the Environment, Laboratory Services Branch is responsible for the analysis of a wide variety of sample types for metals and non-metals. The use of sensitive instrumentation and methodologies appropriate to the sample matrix, combined with quality assurance programs, ensures that the Section is able to maintain a high standard of analytical performance. This performance is monitored through regular internal quality control and assurance programs as well as participation in interlaboratory round-robins. This QA report summarizes the methodologies used for analysis of these samples and the supporting internal quality assurance data.

This report is assembled in sections that reflect the analyses performed on different sample matrices in support of the programs of the Ministry of the Environment. Coincidentally, these divisions also reflect the supervisory responsibilities within the Section.

II. Quality Control and Assurance

The objectives of the quality control and assurance programs are to ensure that all of the components of the analytical process are under control and to ensure immediate detection and correction of unacceptable analytical performance. The program monitors all of the reagents, instrumentation, calibration and recovery components of the analytical system.

A. Quality Control

Quality control of the analytical process takes place at the instrument level and is intended to ensure that the instrumentation is operating according to established criteria. This control function ensures that instrument calibration, standardization, slope and intercept, and instrumental drift meet these criteria.

B. Quality Assurance

Quality assurance of the analytical process takes place after the results have been generated and is intended to ensure that the analytical protocols of sample preparation and digestion have been carried out correctly. This control function ensures that reagent blanks, digested standards, sample duplicates and recovery materials meet established response criteria.

III. Report Format

The report consists of one page method summaries and one page data summaries of blanks, between-run controls and within-run duplicates in formats that are common to all of the parameter/matrix combinations. The method summaries give a brief outline of the sample preparation and measurement procedures. The data summaries consist of annual mean values with standard deviations.

For the within-run duplicates, the data set is subdivided into ranges approximating 0 to 20 %, 20 to 50 % and 50 to 100% of the analytical range. All results for duplicates reported to the data base that are "<" or that have been diluted into the range are excluded from the statistical analysis.

The standard deviations for blanks and between-run controls are calculated using formula I. Formula II is used for the calculations for within-run duplicates.

$$sd = sqrt[\{(sumx2 - (sumx)2\}/n/(n-1)] \quad .....I$$

$$sd = sqrt(sumd2/2n) \quad .....II$$

where : x = the individual values; n = the number of events
d = the differences between pairs of duplicates

The data is stored in a personal computer using BMB Manager II files. All data manipulations, reports generated etc, are performed using applications written in Manager Math.

TABLE OF CONTENTS

9. Sewage

9.1 Sewage Samples

Sewage samples consist of three distinct matrix types: raw sewage, final effluent and sludge. Due to their physical nature and the expected levels of the analytes, each of these sample matrices is prepared and analysed differently.

Table 9.1 summarizes the parameters determined in sewage type samples, the preparation procedure and method of analysis.

TABLE 9.1

Parameter	Collection Device	Preparation	Analysis
Metals	Glass or plastic bottles	Acid digest	AAS,ICP-AES
Mercury	Glass or plastic bottles	Acid digest	Cold vapour AAS
Hydride Metals	Glass or plastic bottles	Acid digest	AAS
Cyanide	Glass or plastic bottles	Distillation	Automated colorimetry

9.2 Sewage Quality Assurance

Sample duplicates are generated by aliquoting two separate portions of the samples.

Sewage QA samples are composited real samples where possible and standard solutions in other cases.

Table 9.2 summarizes the sample designations, their source and use as QA samples for the analysis of sewage type samples.

TABLE 9.2

Sample Designation	Type	Parameter
RF29	Composite dried sludge	Metals, Hg
RF31	Composite raw sewage	Metals, Hg
785	Composite sewage	Hg
475-3	EPA standard solution No 475	Hydrides
swc1,c2	Composite sewage	Hydrides
qca1,b1,qcd	Standard solutions	Cyanide

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Aluminum TEST CODE:ALUT SAMPLE TYPE:Raw Sewage
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 300 ml
Container- Glass or plastic bottle (500 ml)
Preservative- 10 drops of conc. HNO3 to 500 ml.
Other- Fill only 2/3rd of the container

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted-100%
Procedure- Transfer 25 ml of a well mixed sample to a test tube marked at 25 ml volume. Dry overnight in a forced air oven at 105°C. Add 1 ml of HNO3 and 3 ml HCl and digest for about 6 hrs. The volume remaining should be less than 2 ml.
Add distilled water to the 25 ml mark and with distilled water and mix well. Allow particles to settle or if necessary filter through a Whatman #40 filter paper. Determine the elemental concentration by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 100 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.5 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.008 - 2.0 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	4.0 mg/L	
std. dev.	0.3 mg/L	
R.S.D.	6.9 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.4	0.2	0.2
mean	0.3	0.7	1.3

W 0.1 mg/L T 0.5 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref 31) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM IN RAW SEWAGE

Operating Range = 0.008 to 2.0 mg/L

IN - RUN DUPLICATES

range	<0.008	0.008 to0.40	0.40 to1.00	1.00 to2.0	>2.0
no.	25	6	22	18	31
s.w.		0.3914	0.1545	0.1588	
mean		0.2760	0.7330	1.3340	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	107	3.957	0.2745	6.94

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	17	0.645	0.0905

DATE 87/02/28

TEST NAME:Arsenic TEST CODE:ASUT SAMPLE TYPE:Liq Sldge/Sew
UNIT: Biomaterials SUPERVISOR: R. S. Sadana

METHOD CODE: TYPE: Semi-aut. hydr. gen - flameless AAS
REVISION NO: Original DATE: January, 1983
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 10 ml
Container- Glass bottle with bakelite screw cap (16 oz)
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted->90
Procedure- Pipette 1 ml of sample into a 18 x 150 mm pyrex graduated test tube. Add 3 ml of acid mixture (6 nitric: 3 sulphuric: 1 perchloric). Process in batches of 80 samples including blanks, calibration standards and controls.
Digest in an aluminum hot block at a medium setting on the hot plate for 14 hrs until dense white fumes appear.
Cool,add 0.5 ml of distilled water, then 2 ml conc. HCl.
Dilute to 15 ml with distilled water and mix.
Feed the prepared solutions to the automated system for the determination of arsenic by the hydride-FAAS technique.
INTERFERENCES: Excessive concentrations of Cu, Fe and Ni may interfere.
REPORTING RESULTS:2 dec. for <10, 1 dec.<100, whole no. if >100 µg/ml
INSTRUMENTATION: Atomic absorption spectrophotometer (Varian Techtron 1200,with strip chart recorder, peristaltic pump and autosampler (Technicon or Gilson).Open ended quartz "T" atomizer (0.6x10cm);gas-
Calibration Range:0 - 40 ng/ml (linear<20ng/ml) /liquid separator)
Resolution: 0.01 absorbance (unexpanded scale)
Sensitivity:0.02 ug/ml reads 0.15 abs.
Instrument Detection Limit: 0.001 ug/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.01 - 0.6 mg/L
Accuracy- 99% (EPA Standard)
Precision of Controls-

	A	B
mean	.106 mg/L	.206
std. dev.	.010 mg/L	.011
R.S.D.	9.7 %	5.3 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.004	0.012	0.051
mean	0.072	0.208	0.369

W 0.01 mg/L T 0.05 mg/L

CONTROL LIMITS:

REMARKS:

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

ARSENIC IN SEWAGE

Operating Range = 0.010 to 0.6 mg/L

IN - RUN DUPLICATES

range	<0.010	0.010 to0.12	0.12 to0.30	0.30 to0.6	>0.6
no.	60	18	29	10	8
s.w.		0.0037	0.0117	0.0511	
mean		0.0720	0.2080	0.3690	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
swc1	53	0.106	0.0103	9.72
swc2	51	0.206	0.0110	5.34
475-3	53	0.405	0.0161	3.98

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/02/27

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Cadmium TEST CODE:CDUT SAMPLE TYPE:Raw Sewage
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 300 ml
Container- Glass or plastic bottle (500 ml)
Preservative- 10 drops of conc. HNO3 to 500 ml.
Other- Fill only 2/3rd of the container

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted-100%
Procedure- Transfer 25 ml of a well mixed sample to a test tube marked at 25 ml volume. Dry overnight in a forced air oven at 105°C. Add 1 ml of HNO3 and 3 ml HCl and digest for about 6 hrs. The volume remaining should be less than 2 ml.
Add distilled water to the 25 ml mark and with distilled water and mix well. Allow particles to settle or if necessary filter through a Whatman #40 filter paper. Determine the elemental concentration by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 0.4 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.01 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.001 - 0.10 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	0.20 mg/L	
std. dev.	0.01 mg/L	
R.S.D.	6.9 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.008	0.002	
mean	0.009	0.031	

W .005 mg/L T .025 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref QCFE1) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.
  - assumed 100%.
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM IN RAW SEWAGE

Operating Range = 0.001 to 0.1 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.02	0.02 to0.05	0.05 to0.1	>0.1
no.	86	13	6	0	7
s.w.		0.0084	0.0023	0.0000	
mean		0.0090	0.0310	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	117	0.196	0.0135	6.89

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	5	0.013	0.0036

DATE 87/02/28

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Cobalt TEST CODE:COUT SAMPLE TYPE:Raw Sewage
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 300 ml
Container- Glass or plastic bottle (500 ml)
Preservative- 10 drops of conc. HNO3 to 500 ml.
Other- Fill only 2/3rd of the container

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted-100%
Procedure- Transfer 25 ml of a well mixed sample to a test tube marked at 25 ml volume. Dry overnight in a forced air oven at 105°C. Add 1 ml of HNO3 and 3 ml HCl and digest for about 6 hrs. The volume remaining should be less than 2 ml.
Add distilled water to the 25 ml mark and with distilled water and mix well. Allow particles to settle or if necessary filter through a Whatman #40 filter paper. Determine the elemental concentration by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 0.4 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.02 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range-0.002 to 0.1 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	.084 mg/L	
std. dev.	.015 mg/L	
R.S.D.	18.1 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.019	0.010	
mean	0.013	0.032	

W 0.02 mg/L T 0.10 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref 31) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.
  - assumed 100%.
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

COBALT IN RAW SEWAGE

Operating Range = 0.002 to 0.1 mg/L

IN - RUN DUPLICATES

range	<0.002	0.002 to0.02	0.02 to0.05	0.05 to0.1	>0.1
no.	70	22	18	0	2
s.w.		0.0191	0.0103	0.0000	
mean		0.0130	0.0320	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	117	0.084	0.0152	18.10

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	21	0.029	0.0098

DATE 87/02/28

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Copper TEST CODE:CUUT SAMPLE TYPE:Raw Sewage
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 300 ml
Container- Glass or plastic bottle (500 ml)
Preservative- 10 drops of conc. HNO3 to 500 ml.
Other- Fill only 2/3rd of the container

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted-100%
Procedure- Transfer 25 ml of a well mixed sample to a test tube marked at 25 ml volume. Dry overnight in a forced air oven at 105°C. Add 1 ml of HNO3 and 3 ml HCl and digest for about 6 hrs. The volume remaining should be less than 2 ml.
Add distilled water to the 25 ml mark and with distilled water and mix well. Allow particles to settle or if necessary filter through a Whatman #40 filter paper. Determine the elemental concentration by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2.0 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.02 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.001 to 1.0 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	1.58 mg/L	
std. dev.	0.09 mg/L	
R.S.D.	5.6 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.010	0.107	0.029
mean	0.079	0.329	0.580

W 0.01 mg/L T 0.05 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref 31) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER IN RAW SEWAGE

Operating Range = 0.001 to 1.0 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.20	0.20 to0.50	0.50 to1.0	>1.0
no.	15	61	14	5	7
s.w.		0.0095	0.1070	0.0291	
mean		0.0790	0.3290	0.5800	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	107	1.582	0.0890	5.63

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	0	0.000	0.0000

DATE 87/02/28

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Chromium TEST CODE:CRUT SAMPLE TYPE:Raw Sewage
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 300 ml
Container- Glass or plastic bottle (500 ml)
Preservative- 10 drops of conc. HNO3 to 500 ml.
Other- Fill only 2/3rd of the container

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted-100%
Procedure- Transfer 25 ml of a well mixed sample to a test tube marked at 25 ml volume. Dry overnight in a forced air oven at 105°C. Add 1 ml of HNO3 and 3 ml HCl and digest for about 6 hrs. The volume remaining should be less than 2 ml.
Add distilled water to the 25 ml mark and with distilled water and mix well. Allow particles to settle or if necessary filter through a Whatman #40 filter paper. Determine the elemental concentration by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2.0 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.03 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.001 - 0.10 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	5.62 mg/L	
std. dev.	0.44 mg/L	
R.S.D.	7.8 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.02	0.02	0.04
mean	0.02	0.04	0.07

W 0.02 mg/L T 0.10 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref 31) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

CHROMIUM IN RAW SEWAGE

Operating Range = 0.001 to 0.1 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.02	0.02 to0.05	0.05 to0.1	>0.1
no.	48	8	8	9	29
s.w.		0.0244	0.0193	0.0443	
mean		0.0170	0.0410	0.0730	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	116	5.623	0.4394	7.81

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	5	0.041	0.0145

DATE 87/02/28

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Iron TEST CODE:FEUT SAMPLE TYPE:Raw Sewage
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 300 ml
Container- Glass or plastic bottle (500 ml)
Preservative- 10 drops of conc. HNO3 to 500 ml.
Other- Fill only 2/3rd of the container

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted-100%
Procedure- Transfer 25 ml of a well mixed sample to a test tube marked at 25 ml volume. Dry overnight in a forced air oven at 105°C. Add 1 ml of HNO3 and 3 ml HCl and digest for about 6 hrs. The volume remaining should be less than 2 ml. Add distilled water to the 25 ml mark and with distilled water and mix well. Allow particles to settle or if necessary filter through a Whatman #40 filter paper. Determine the elemental concentration by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 10 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.5 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.002 - 25.0 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	4.18 mg/L	
std. dev.	0.24 mg/L	
R.S.D.	5.6 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.13	4.20	0.46
mean	1.72	8.07	18.40

W 0.05 mg/L T 0.25 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref 31) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON IN RAW SEWAGE

Operating Range = 0.002 to 25.0 mg/L

IN - RUN DUPLICATES

range	<0.002	0.002 to5.00	5.00 to12.50	12.50 to25.0	>25.0
no.	6	62	20	10	4
s.w.		0.1267	4.1957	0.4595	
mean		1.7180	8.0730	18.3980	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	117	4.184	0.2361	5.64

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	12	0.982	1.0511

DATE 87/01/21

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Lead TEST CODE:PBUT SAMPLE TYPE:Raw Sewage
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 300 ml
Container- Glass or plastic bottle (500 ml)
Preservative- 10 drops of conc. HNO3 to 500 ml.
Other- Fill only 2/3rd of the container

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted-100%
Procedure- Transfer 25 ml of a well mixed sample to a test tube marked at 25 ml volume. Dry overnight in a forced air oven at 105°C. Add 1 ml of HNO3 and 3 ml HCl and digest for about 6 hrs. The volume remaining should be less than 2 ml.
Add distilled water to the 25 ml mark and with distilled water and mix well. Allow particles to settle or if necessary filter through a Whatman #40 filter paper. Determine the elemental concentration by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2.0 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.15 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.02 - 0.20 mg/L1
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	1.14 mg/L	
std. dev.	0.09 mg/L	
R.S.D.	8.2 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.		0.12	0.13
mean		0.08	0.16

W 0.02 mg/L T 0.10 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref 31) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD IN RAW SEWAGE

Operating Range = 0.002 to 0.2 mg/L

IN - RUN DUPLICATES

range	<0.002	0.002 to0.04	0.04 to0.10	0.10 to0.2	>0.2
no.	86	0	6	4	16
s.w.		0.0000	0.1182	0.1333	
mean		0.0000	0.0830	0.1550	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	117	1.135	0.0934	8.23

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	2	0.212	0.0435

DATE 87/02/28

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Manganese TEST CODE:MNUT SAMPLE TYPE:Raw Sewage
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 300 ml
Container- Glass or plastic bottle (500 ml)
Preservative- 10 drops of conc. HNO3
Other- Fill only 2/3rd of the container

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted-100%
Procedure- Transfer 25 ml of a well mixed sample to a test tube marked at 25 ml volume. Dry overnight in a forced air oven at 105°C. Add 1 ml of HNO3 and 3 ml HCl and digest for about 6 hrs. The volume remaining should be less than 2 ml.
Add distilled water to the 25 ml mark and with distilled water and mix well. Allow particles to settle or if necessary filter through a Whatman #40 filter paper. Determine the elemental concentration by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2.0 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit:0.01 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.001 - 0.10 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	.084 mg/L	
std. dev.	.006 mg/L	
R.S.D.	6.6 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.003	0.003	0.019
mean	0.013	0.038	0.074

W .005 mg/L T .025 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref 31) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE IN RAW SEWAGE

Operating Range = 0.001 to 0.1 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.02	0.02 to0.05	0.05 to0.1	>0.1
no.	3	7	14	27	51
s.w.		0.0034	0.0026	0.0188	
mean		0.0130	0.0380	0.0740	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	117	0.084	0.0055	6.55

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	0	0.000	0.0000

DATE 87/02/28

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Mercury TEST CODE:HGUT HGFT SAMPLE TYPE:Sewage sludge
UNIT: Biomaterials SUPERVISOR: R. S. Sadana

METHOD CODE: TYPE: Flameless AAS
REVISION NO: Original DATE: May, 1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 250 ml
Container- Wide mouth glass jar
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pipette 10 ml of sample into a 125 ml Phillips beaker. Add 10 ml of acid mixture (4:3:1 - H2O:HCl:HNO3).
Heat on a hot plate (approx 83°C) for 5 minutes.
Add 36 ml distilled water and 15 ml KMnO4 (saturated) and digest the contents for 45 minutes. Filter the solution and dilute to 100 ml with distilled water.
Treat blanks and calibration standards in exactly the same manner.
Determine mercury in the entire volume. The measurement step is automated and is based on the evolution of atomic vapour of mercury (wavelength - 254nm) by the addition of a reducing agent.
INTERFERENCES: Water vapour; organic solvents.

REPORTING RESULTS:Two significant figures.
INSTRUMENTATION: Automated sampler and peristaltic pump.
Laboratory Data Control U.V. monitor.

Calibration Range: 0 - 20 ng/ml
Resolution: 0.25 ng/ml (one division on recorder chart paper)
Sensitivity:10 ng/100 ml reads 0.2 absorbance (20 divs on chart)
Instrument Detection Limit: 0.1 ng/ml

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.010 - 0.3 mg/L
Accuracy- No standards available
Precision of Controls-

	A	B
mean	.064 mg/L	
std. dev.	.010 mg/L	
R.S.D.	15.6 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0055	0.0134	0.0100
mean	0.029	0.073	0.178

W 0.01 mg/L T 0.05 mg/L

CONTROL LIMITS:

REMARKS:

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

MERCURY IN SEWAGE

Operating Range = 0.010 to 0.3 mg/L

IN - RUN DUPLICATES

range	<0.010	0.010 to0.05	0.05 to0.13	0.13 to0.3	>0.3
no.	9	13	12	5	2
s.w.		0.0055	0.0134	0.0100	
mean		0.0290	0.0730	0.1780	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
785	103	0.064	0.0100	15.63

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	4	0.058	0.0050

DATE 87/01/05

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Molybdenum TEST CODE:MOUT SAMPLE TYPE:Raw Sewage
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 300 ml
Container- Glass or plastic bottle (500 ml)
Preservative- 10 drops of conc. HNO3 to 500 ml.
Other- Fill only 2/3rd of the container

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted-100%
Procedure- Transfer 25 ml of a well mixed sample to a test tube marked at 25 ml volume. Dry overnight in a forced air oven at 105°C. Add 1 ml of HNO3 and 3 ml HCl and digest for about 6 hrs. The volume remaining should be less than 2 ml.
Add distilled water to the 25 ml mark and with distilled water and mix well. Allow particles to settle or if necessary filter through a Whatman #40 filter paper. Determine the elemental concentration by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 0.4 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.05 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.001 - 0.10 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	.425 mg/L	
std. dev.	.047 mg/L	
R.S.D.	11.1 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.018	0.038	0.006
mean	0.013	0.030	0.069

W .002 mg/L T .010 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref 31) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

MOLYBDENUM IN RAW SEWAGE

Operating Range = 0.001 to 0.1 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.02	0.02 to0.05	0.05 to0.1	>0.1
no.	87	1	11	9	4
s.w.		0.0183	0.0381	0.0055	
mean		0.0130	0.0300	0.0690	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	117	0.425	0.0470	11.06

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	17	0.061	0.0127

DATE 87/02/28

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Nickel TEST CODE:NIUT SAMPLE TYPE:Raw Sewage
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 300 ml
Container- Glass or plastic bottle (500 ml)
Preservative- 10 drops of conc. HNO3 to 500 ml.
Other- Fill only 2/3rd of the container

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted-100%
Procedure- Transfer 25 ml of a well mixed sample to a test tube marked at 25 ml volume. Dry overnight in a forced air oven at 105°C. Add 1 ml of HNO3 and 3 ml HCl and digest for about 6 hrs. The volume remaining should be less than 2 ml.
Add distilled water to the 25 ml mark and with distilled water and mix well. Allow particles to settle or if necessary filter through a Whatman #40 filter paper. Determine the elemental concentration by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2.0 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.06 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.003 - 0.1 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	1.28 mg/L	
std. dev.	0.07 mg/L	
R.S.D.	5.6 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.		0.05	0.01
mean		0.04	0.08

W 0.02 mg/L T 0.10 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref 31) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency assumed to be 100% as material originally in solution. No reference standards available.
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL IN RAW SEWAGE

Operating Range = 0.003 to 0.1 mg/L

IN - RUN DUPLICATES

range	<0.003	0.003 to0.02	0.02 to0.05	0.05 to0.1	>0.1
no.	81	0	9	4	18
s.w.		0.0000	0.0498	0.0092	
mean		0.0000	0.0350	0.0800	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	117	1.282	0.0712	5.55

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	7	0.083	0.0193

DATE 87/02/28

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Selenium TEST CODE:SEUT SAMPLE TYPE:Liq Sldge/Sew
UNIT: Biomaterials SUPERVISOR: R. S. Sadana

METHOD CODE: TYPE: Semi-aut. hydr. gen - flameless AAS
REVISION NO: Original DATE: January, 1983
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 10 ml
Container- Glass bottle with bakelite screw cap (16 oz)
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted->90
Procedure- Pipette 1 ml of sample into a 18 x 150 mm pyrex graduated test tube. Add 3 ml of acid mixture (6 nitric: 3 sulphuric: 1 perchloric). Process in batches of 80 samples including blanks, calibration standards and controls.
Digest in an aluminum hot block at a medium setting on the hot plate for 14 hrs until dense white fumes appear.
Cool,add 0.5 ml of distilled water, then 2 ml conc. HCl.
Dilute to 15 ml with distilled water and mix.
Feed the prepared solutions to the automated system for the determination of selenium by the hydride-FAAS technique.
INTERFERENCES: Excessive concentrations of Cu, Fe and Ni may interfere.
REPORTING RESULTS:2 dec. for <10, 1 dec.<100, 0 dec. if >100 µg/ml
INSTRUMENTATION: Atomic absorption spectrophotometer (Varian Techtron 1200,with strip chart recorder, peristaltic pump and autosampler (Technicon or Gilson).Open ended quartz "T" atomizer (0.6x10cm);gas-
Calibration Range:0 - 40 ng/ml (linear<20ng/ml) /liquid separator)
Resolution: 0.01 absorbance (unexpanded scale)
Sensitivity:0.02 ug/ml reads 0.20 abs.
Instrument Detection Limit: 0.001 ug/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.01 - 0.6 mg/L
Accuracy- 99% (EPA Standard)
Precision of Controls-

	A	B
mean	.066 mg/L	.167
std. dev.	.005	.008
R.S.D.	7.9 %	5.0 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.009	0.015	0.041
mean	0.083	0.164	0.372

W 0.01 mg/L T 0.05 mg/L

CONTROL LIMITS:

REMARKS:

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

SELENIUM IN SEWAGE

Operating Range = 0.010 to 0.6 mg/L

IN - RUN DUPLICATES

range	<0.010	0.010 to0.12	0.12 to0.30	0.30 to0.6	>0.6
no.	64	31	22	3	2
s.w.		0.0087	0.0145	0.0410	
mean		0.0830	0.1640	0.3720	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
swc1	42	0.066	0.0052	7.88
swc2	43	0.167	0.0084	5.03
475-3	51	0.069	0.0050	7.25

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/03/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Zinc TEST CODE:ZNUT SAMPLE TYPE:Raw Sewage
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 300 ml
Container- Glass or plastic bottle (500 ml)
Preservative- 10 drops of conc. HNO3 to 500 ml.
Other- Fill only 2/3rd of the container

SAMPLE PREPARATION:Partial Extn.- Total Extn.-yes % Extracted-100%
Procedure- Transfer 25 ml of a well mixed sample to a test tube marked at 25 ml volume. Dry overnight in a forced air oven at 105°C. Add 1 ml of HNO3 and 3 ml HCl and digest for about 6 hrs. The volume remaining should be less than 2 ml.
Add distilled water to the 25 ml mark and with distilled water and mix well. Allow particles to settle or if necessary filter through a Whatman #40 filter paper. Determine the elemental concentration by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2.0 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.02 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.001 - 1.0 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	0.70 mg/L	
std. dev.	0.10 mg/L	
R.S.D.	13.3 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.04	0.02	0.17
mean	0.09	0.31	0.74

W 0.02 mg/L T 0.10 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref 31) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC IN RAW SEWAGE

Operating Range = 0.001 to 1.0 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.20	0.20 to0.50	0.50 to1.0	>1.0
no.	5	63	20	6	18
s.w.		0.0336	0.0182	0.1748	
mean		0.0940	0.3090	0.7370	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	117	0.701	0.0973	13.88

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	31	0.032	0.0113

DATE 87/02/28

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Total cyanide TEST CODE: CCNAUR SAMPLE TYPE:Sewage
UNIT: QC-Project SUPERVISOR: J. Hipfner

METHOD CODE:001AC2
REVISION NO: DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Glass or plastic (preferred)
Preservative- NaOH
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.- % Extracted-100
Procedure- The sample is first run to see if there is cyanide present. The is run directly by the automated high temperature distillation with 25% H3PO4-5% H3PO2 followed by a colourimetric analysis with chloramine T -isonicotinic acid -barbituric acid method.
If the total cyanide is > .01 mg/L then 5 to 250 ml of sample is manually distilled with 30 ml of 15%(w/v)tartaric acid. The distillate is collected in 50 ml of 1N NaOH, and analyzed by the automated Technicon distillation system referred to above.

INTERFERENCES: SCN interference is removed by distillation. Distillable organics may interfere; also S= at high levels.
REPORTING RESULTS:Mg/l CN: 3 decimal places up to 3 significant figs
INSTRUMENTATION: Technicon AAII continuous flow analyzer including pump, colourimeter, appropriate autosampler and recorder. High temperature distillation apparatus (Technicon). Manual dist. app
Calibration Range: 0 to 0.4 mg/l as CN
Resolution: 0.001 mg/l
Sensitivity:
Instrument Detection Limit: 0.001 mg/l
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.00100 to 0.400 mg/l
Accuracy- 100%
Precision of Controls-

	A	B
mean	.110 mg/L	0.059
std. dev.	.0027mg/L	0.0026
R.S.D.	2.45 %	4.41 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0011	0.0009	0.0127
mean	0.014	0.119	0.293

W .001 mg/L T .005 mg/L

CONTROL LIMITS:

REMARKS: Pure CN standards are recovered 100% during manual distillation. Complex cyanides can normally be expected to be recovered at 100%.

SUMMARY REPORT OF QUALITY CONTROL DATA

TOTAL CYANIDE IN SEWAGE

Operating Range = 0.001 to 0.4 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.08	0.08 to0.2	0.2 to0.4	>0.4
no.	0	44	2	0	0
s.w.		0.0009	0.0041	0	
mean		0.0141	0.089	0	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qc-a	146	0.149	0.0049	3.29
qc-b	146	0.018	0.0022	12.22

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	146	0.001	0

DATE 88/06/01

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Free cyanide TEST CODE: CCNFUR SAMPLE TYPE: Sewage
UNIT: QC-Project SUPERVISOR: J. Hipfner

METHOD CODE:700AC2
REVISION NO: DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 100 ml
Container- Glass or plastic (preferred)
Preservative- NaOH
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.- % Extracted- *
Procedure- Pass sample aliquot through an automated low temperature distillation (106°C) in a distillation acid consisting of 10% acetic acid and 0.5% zinc acetate.
Analyze distillate by the Chloramine-T -pyridine-barbituric acid colourometric method, or equivalent.

INTERFERENCES: None

REPORTING RESULTS:Mg/l CN to 2 decimal places up to 3 significant figs
INSTRUMENTATION: Technicon automated continuous flow analyzer including pump, colourimetric distillation apparatus and sampler; suitable recorder.
Calibration Range: 0 to 0.4 mg/l as CN
Resolution: 0.001
Sensitivity:
Instrument Detection Limit: 0.001 mg/l
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0100 to 0.400 mg/L
Accuracy- 100%
Precision of Controls-

	A	B
mean	.110 mg/L	0.060
std. dev.	.0036mg/L	0.0031
R.S.D.	3.27 %	5.17%

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0004	0.0007	0.0000
mean	0.0135	0.098	0.290

W .001 mg/L T .005 mg/L

CONTROL LIMITS:

REMARKS:* The test defines the results reported in this case. The terminology "Weak Acid Dissociable" is commonly used and represents weakly associated cyanide compounds such as KCN, NaCN, NiCN4, HCN,etc.

SUMMARY REPORT OF QUALITY CONTROL DATA

FREE CYANIDE IN SEWAGE

Operating Range = 0.001 to 0.4 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.08	0.08 to0.2	0.2 to0.4	>0.4
no.	0	44	1	1	0
s.w.		0.0011	0.0141	0.0078	
mean		0.0069	0.13	0.3485	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qc-a	135	0.151	0.0062	4.11
qc-b	135	0.018	0.0022	12.22

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	135	0.001	0

DATE 88/06/01

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Aluminum TEST CODE:ALUT SAMPLE TYPE:Final Efflnt
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Glass or polyethylene bottle (1 litre)
Preservative- 10 drops of conc. HNO3 in 500 ml of sample.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-87.5%
Procedure- Transfer 50 ml of a well mixed sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml of HNO3 and dry in a forced air oven at 105 °C. Add 2 ml of aqua-regia and digest for 2 hrs.

Cool, and make to final volume of 12.5 ml with distilled water. Allow time for particles to settle and analyze by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 100 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.5 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.5 - 1000 mg/l
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	3.87 mg/L	
std. dev.	0.22 mg/L	
R.S.D.	5.6 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.024	0.027	0.006
mean	0.204	0.649	1.050

W 0.01 mg/L T 0.05 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref QCFE1) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency measured by spike recovery.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM IN FINAL EFFLUENT

Operating Range = 0.008 to 2.0 mg/L

IN - RUN DUPLICATES

range	<0.008	0.008 to0.40	0.40 to1.00	1.00 to2.0	>2.0
no.	7	20	11	1	6
s.w.		0.0237	0.0270	0.0064	
mean		0.2040	0.6490	1.0500	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	31	3.867	0.2184	5.65

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	4	0.492	0.0912

DATE 87/01/21

Inorganic Trace Contaminants Section

TEST NAME:Arsenic TEST CODE:ASUT SAMPLE TYPE:Liq Sldge/Sew
UNIT: Biomaterials SUPERVISOR: R. S. Sadana

METHOD CODE: TYPE: Semi-aut. hydr. gen - flameless AAS
REVISION NO: Original DATE: January, 1983
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 10 ml
Container- Glass bottle with bakelite screw cap (16 oz)
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted->90
Procedure- Pipette 1 ml of sample into a 18 x 150 mm pyrex graduated test tube. Add 3 ml of acid mixture (6 nitric: 3 sulphuric: 1 perchloric). Process in batches of 80 samples including blanks, calibration standards and controls.
Digest in an aluminum hot block at a medium setting on the hot plate for 14 hrs until dense white fumes appear.
Cool,add 0.5 ml of distilled water, then 2 ml conc. HCl.
Dilute to 15 ml with distilled water and mix.
Feed the prepared solutions to the automated system for the determination of arsenic by the hydride-FAAS technique.
INTERFERENCES: Excessive concentrations of Cu, Fe and Ni may interfere.
REPORTING RESULTS:2 dec. for <10, 1 dec.<100, whole no. if >100 µg/ml
INSTRUMENTATION: Atomic absorption spectrophotometer (Varian Techtron 1200,with strip chart recorder, peristaltic pump and autosampler (Technicon or Gilson).Open ended quartz "T" atomizer (0.6x10cm);gas-liquid separator)
Calibration Range:0 - 40 ng/ml (linear<20ng/ml)
Resolution: 0.01 absorbance (unexpanded scale)
Sensitivity:0.02 ug/ml reads 0.15 abs.
Instrument Detection Limit: 0.001 ug/ml

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.01 - 0.6 mg/L
Accuracy- 99% (EPA Standard)
Precision of Controls-

	A	B
mean	.106 mg/L	.206
std. dev.	.010 mg/L	.011
R.S.D.	9.7 %	5.3 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.004	0.012	0.051
mean	0.072	0.208	0.369

W 0.01 mg/L T 0.05 mg/L

CONTROL LIMITS:

REMARKS:

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

ARSENIC IN SEWAGE

Operating Range = 0.010 to 0.6 mg/L

IN - RUN DUPLICATES

range	<0.010	0.010 to0.12	0.12 to0.30	0.30 to0.6	>0.6
no.	60	18	29	10	8
s.w.		0.0037	0.0117	0.0511	
mean		0.0720	0.2080	0.3690	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
swc1	53	0.106	0.0103	9.72
swc2	51	0.206	0.0110	5.34
475-3	53	0.405	0.0161	3.98

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/02/27

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Cadmium TEST CODE:CDUT SAMPLE TYPE:Final Efflnt
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Glass or polyethylene bottle (1 litre)
Preservative- 10 drops of conc. HNO3 in 500 ml of sample.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-88.5%
Procedure- Transfer 50 ml of a well mixed sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml of HNO3 and dry in a forced air oven at 105 °C. Add 2 ml of aqua-regia and digest for 2 hrs.

Cool, and make to final volume of 12.5 ml with distilled water. Allow time for particles to settle and analyze by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 0.4 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.01 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.001 - 0.1 mg/l
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	.180 mg/L	
std. dev.	.033 mg/L	
R.S.D.	18.6 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.002		
mean	0.003		

W .001 mg/L T .005 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref QCFE1) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency measured by spike recovery.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM IN FINAL EFFLUENT

Operating Range = 0.001 to 0.1 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.02	0.02 to0.05	0.05 to0.1	>0.1
no.	30	21	0	0	0
s.w.		0.0016	0.0000	0.0000	
mean		0.0030	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	36	0.180	0.0334	18.56

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	6	0.013	0.0031

DATE 87/01/21

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Chromium TEST CODE:CRUT SAMPLE TYPE:Final Efflnt
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Glass or polyethylene bottle (1 litre)
Preservative- 10 drops of conc. HNO3 in 500 ml of sample.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-89%
Procedure- Transfer 50 ml of a well mixed sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml of HNO3 and dry in a forced air oven at 105 °C. Add 2 ml of aqua-regia and digest for 2 hrs.

Cool, and make to final volume of 12.5 ml with distilled water. Allow time for particles to settle and analyze by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.03 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.001 - 0.10 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	5.26 mg/L	
std. dev.	1.26 mg/L	
R.S.D.	24.0 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.006	0.040	0.021
mean	0.008	0.036	0.064

W .002 mg/L T .010 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref QCFE1) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency measured by spike recovery.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

CHROMIUM IN FINAL EFFLUENT

Operating Range = 0.001 to 0.1 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.02	0.02 to0.05	0.05 to0.1	>0.1
no.	24	4	3	3	11
s.w.		0.0058	0.0399	0.0214	
mean		0.0080	0.0360	0.0640	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	35	5.259	1.2596	23.95

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	3	0.045	0.0045

DATE 87/01/21

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Cobalt TEST CODE:COUT SAMPLE TYPE:Final Efflnt
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Glass or polyethylene bottle (1 litre)
Preservative- 10 drops of conc. HNO3 in 500 ml of sample.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-91%
Procedure- Transfer 50 ml of a well mixed sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml of HNO3 and dry in a forced air oven at 105 °C. Add 2 ml of aqua-regia and digest for 2 hrs.

Cool, and make to final volume of 12.5 ml with distilled water. Allow time for particles to settle and analyze by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 0.4 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.02 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.002 - 0.10 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	.079 mg/L	
std. dev.	.014 mg/L	
R.S.D.	17.2 %	

Precision of Duplicates-low range mid range high range

s.d.	0.004	
mean	0.005	

W .002 mg/L T .010 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref QCFE1) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency measured by spike recovery.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

COBALT IN FINAL EFFLUENT

Operating Range = 0.002 to 0.1 mg/L

IN - RUN DUPLICATES

range	<0.002	0.002 to0.02	0.02 to0.05	0.05 to0.1	>0.1
no.	30	20	0	0	1
s.w.		0.0037	0.0000	0.0000	
mean		0.0050	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	36	0.079	0.0136	17.22

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	18	0.032	0.0098

DATE 87/01/21

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Copper TEST CODE:CUUT SAMPLE TYPE:Final Efflnt
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Glass or polyethylene bottle (1 litre)
Preservative- 10 drops of conc. HNO3 in 500 ml of sample.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-98.5%
Procedure- Transfer 50 ml of a well mixed sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml of HNO3 and dry in a forced air oven at 105 °C. Add 2 ml of aqua-regia and digest for 2 hrs.

Cool, and make to final volume of 12.5 ml with distilled water. Allow time for particles to settle and analyze by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2.0 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.02 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range-0.001 to 1.0 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	1.52 mg/L	
std. dev.	0.22 mg/L	
R.S.D.	14.8 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.01		0.04
mean	0.03		0.71

W .002 mg/L T .010 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref QCFE1) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency assumed to be 100% as material originally in solution. No reference standards available.
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER IN FINAL EFFLUENT

Operating Range = 0.001 to 1.0 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.20	0.20 to0.50	0.50 to1.0	>1.0
no.	9	30	0	2	4
s.w.		0.0102	0.0000	0.0385	
mean		0.0330	0.0000	0.7090	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	33	1.519	0.2242	14.76

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	11	0.052	0.0187

DATE 87/01/21

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Iron TEST CODE:FEUT SAMPLE TYPE:Final Efflnt
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Glass or polyethylene bottle (1 litre)
Preservative- 10 drops of conc. HNO3 in 500 ml of sample.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-83.5%
Procedure- Transfer 50 ml of a well mixed sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml of HNO3 and dry in a forced air oven at 105 °C. Add 2 ml of aqua-regia and digest for 2 hrs.

Cool, and make to final volume of 12.5 ml with distilled water. Allow time for particles to settle and analyze by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 100 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.5 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.002 - 1.0 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	3.81 mg/L	
std. dev.	0.72 mg/L	
R.S.D.	19.0 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.040	0.095	0.033
mean	0.139	0.294	0714

W .005 mg/L T .025 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref QCFE1) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency measured by spike recovery.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON IN FINAL EFFLUENT

Operating Range = 0.002 to 1.0 mg/L

IN - RUN DUPLICATES

range	<0.002	0.002 to0.20	0.20 to0.50	0.50 to1.0	>1.0
no.	8	12	9	10	6
s.w.		0.0397	0.0950	0.0334	
mean		0.1390	0.2940	0.7140	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	34	3.807	0.7231	18.99

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	6	0.390	0.0728

DATE 87/01/21

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Lead TEST CODE:PBUT SAMPLE TYPE:Final Efflnt
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Glass or polyethylene bottle (1 litre)
Preservative- 10 drops of conc. HNO3 in 500 ml of sample.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-90%
Procedure- Transfer 50 ml of a well mixed sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml of HNO3 and dry in a forced air oven at 105 °C. Add 2 ml of aqua-regia and digest for 2 hrs.

Cool, and make to final volume of 12.5 ml with distilled water. Allow time for particles to settle and analyze by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.15 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.002 - 0.20 mg/l
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	1.00 mg/L	
std. dev.	0.23 mg/L	
R.S.D.	23.0 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.023	0.008	0.014
mean	0.016	0.083	0.115

W 0.02 mg/L T 0.10 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref QCFE1) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency measured by spike recovery.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD IN FINAL EFFLUENT

Operating Range = 0.002 to 0.2 mg/L

IN - RUN DUPLICATES

range	<0.002	0.002 to0.04	0.04 to0.10	0.10 to0.2	>0.2
no.	40	4	2	2	3
s.w.		0.0229	0.0082	0.0142	
mean		0.0160	0.0830	0.1150	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	35	1.000	0.2317	23.17

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	0	0.000	0.0000

DATE 87/01/21

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Manganese TEST CODE:MNUT SAMPLE TYPE:Final Efflnt
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Glass or polyethylene bottle (1 litre)
Preservative- 10 drops of conc. HNO3 in 500 ml of sample
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Transfer 50 ml of a well mixed sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml of HNO3 and dry in a forced air oven at 105 °C. Add 2 ml of aqua-regia and digest for 2 hrs.

Cool, and make to final volume of 12.5 ml with distilled water. Allow time for particles to settle and analyze by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2.0 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.01 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.001 - 0.10 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	.080 mg/L	
std. dev.	.009 mg/L	
R.S.D.	10.9 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.001	0.003	0.005
mean	0.014	0.031	0.069

W .001 mg/L T .005 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref QCFE1) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency measured by spike recovery.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE IN FINAL EFFLUENT

Operating Range = 0.001 to 0.1 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.02	0.02 to0.05	0.05 to0.1	>0.1
no.	1	15	18	7	4
s.w.		0.0007	0.0033	0.0053	
mean		0.0140	0.0310	0.0690	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	35	0.080	0.0087	10.88

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	0	0.000	0.0000

DATE 87/01/21

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Mercury TEST CODE:HGUT HGFT SAMPLE TYPE:Sewage sludge
UNIT: Biomaterials SUPERVISOR: R. S. Sadana

METHOD CODE: TYPE: Flameless AAS
REVISION NO: Original DATE: May, 1984
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 250 ml
Container- Wide mouth glass jar
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-
Procedure- Pipette 10 ml of sample into a 125 ml Phillips beaker. Add 10 ml of acid mixture (4:3:1 - H2O:HCl:HNO3).
Heat on a hot plate (approx 83°C) for 5 minutes.
Add 36 ml distilled water and 15 ml KMnO4 (saturated) and digest the contents for 45 minutes. Filter the solution and dilute to 100 ml with distilled water.
Treat blanks and calibration standards in exactly the same manner.
Determine mercury in the entire volume. The measurement step is automated and is based on the evolution of atomic vapour of mercury (wavelength - 254nm) by the addition of a reducing agent.
INTERFERENCES: Water vapour; organic solvents.

REPORTING RESULTS:Two significant figures.
INSTRUMENTATION: Automated sampler and peristaltic pump. Laboratory Data Control U.V. monitor.

Calibration Range: 0 - 20 ng/ml
Resolution: 0.25 ng/ml (one division on recorder chart paper)
Sensitivity:10 ng/100 ml reads 0.2 absorbance (20 divs on chart)
Instrument Detection Limit: 0.1 ng/ml

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.010 - 0.3 mg/L
Accuracy- No standards available
Precision of Controls-

	A	B
mean	.064 mg/L	
std. dev.	.010 mg/L	
R.S.D.	15.6 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0055	0.0134	0.0100
mean	0.029	0.073	0.178

W 0.01 mg/L T 0.05 mg/L

CONTROL LIMITS:

REMARKS:

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

MERCURY IN SEWAGE

Operating Range = 0.010 to 0.3 mg/L

IN - RUN DUPLICATES

range	<0.010	0.010 to0.05	0.05 to0.13	0.13 to0.3	>0.3
no.	9	13	12	5	2
s.w.		0.0055	0.0134	0.0100	
mean		0.0290	0.0730	0.1780	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
785	103	0.064	0.0100	15.63

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	4	0.058	0.0050

DATE 87/01/05

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Molybdenum TEST CODE:MOUT SAMPLE TYPE:Final Efflnt
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Glass or polyethylene bottle (1 litre)
Preservative- 10 drops of conc. HNO3 in 500 ml of sample.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-95.2%
Procedure- Transfer 50 ml of a well mixed sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml of HNO3 and dry in a forced air oven at 105 °C. Add 2 ml of aqua-regia and digest for 2 hrs.

Cool, and make to final volume of 12.5 ml with distilled water. Allow time for particles to settle and analyze by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 0.4 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.05 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.001 - 0.10 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	.389 mg/L	
std. dev.	.089 mg/L	
R.S.D.	22.8 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.006	0.007	0.001
mean	0.008	0.027	0.059

W .001 mg/L T .005 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref QCFE1) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency measured by spike recovery.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

MOLYBDENUM IN FINAL EFFLUENT

Operating Range = 0.001 to 0.1 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.02	0.02 to0.05	0.05 to0.1	>0.1
no.	37	10	2	1	1
s.w.		0.0064	0.0070	0.0013	
mean		0.0080	0.0270	0.0590	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	35	0.389	0.0885	22.75

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	10	0.054	0.0240

DATE 87/01/21

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Nickel TEST CODE:NIUT SAMPLE TYPE:Final Efflnt
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Glass or polyethylene bottle (1 litre)
Preservative- 10 drops of conc. HNO3 in 500 ml of sample.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-92%
Procedure- Transfer 50 ml of a well mixed sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml of HNO3 and dry in a forced air oven at 105 °C. Add 2 ml of aqua-regia and digest for 2 hrs.

Cool, and make to final volume of 12.5 ml with distilled water. Allow time for particles to settle and analyze by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.06 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.003 - 0.1 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	1.17 mg/L	
std. dev.	0.23 mg/L	
R.S.D.	20.0 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.011	0.005	0.043
mean	0.010	0.029	0.075

W .005 mg/L T .025 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref QCFE1) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency measured by spike recovery.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL IN FINAL EFFLUENT

Operating Range = 0.003 to 0.1 mg/L

IN - RUN DUPLICATES

range	<0.003	0.003 to0.02	0.02 to0.05	0.05 to0.1	>0.1
no.	26	6	3	6	10
s.w.		0.0108	0.0051	0.0427	
mean		0.0100	0.0290	0.0750	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	36	1.167	0.2285	19.58

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	0	0.000	0.0000

DATE 87/01/21

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Selenium TEST CODE:SEUT SAMPLE TYPE:Liq Sldge/Sew
UNIT: Biomaterials SUPERVISOR: R. S. Sadana

METHOD CODE: TYPE: Semi-aut. hydr. gen - flameless AAS
REVISION NO: Original DATE: January, 1983
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- Approximately 10 ml
Container- Glass bottle with bakelite screw cap (16 oz)
Preservative- None
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted->90
Procedure- Pipette 1 ml of sample into a 18 x 150 mm pyrex graduated test tube. Add 3 ml of acid mixture (6 nitric: 3 sulphuric: 1 perchloric). Process in batches of 80 samples including blanks, calibration standards and controls.
Digest in an aluminum hot block at a medium setting on the hot plate for 14 hrs until dense white fumes appear.
Cool,add 0.5 ml of distilled water, then 2 ml conc. HCl.
Dilute to 15 ml with distilled water and mix.
Feed the prepared solutions to the automated system for the determination of selenium by the hydride-FAAS technique.
INTERFERENCES: Excessive concentrations of Cu, Fe and Ni may interfere.
REPORTING RESULTS:2 dec. for <10, 1 dec.<100, 0 dec. if >100 µg/ml
INSTRUMENTATION: Atomic absorption spectrophotometer (Varian Techtron 1200,with strip chart recorder, peristaltic pump and autosampler (Technicon or Gilson).Open ended quartz "T" atomizer (0.6x10cm);gas-
Calibration Range:0 - 40 ng/ml (linear<20ng/ml) /liquid separator)
Resolution: 0.01 absorbance (unexpanded scale)
Sensitivity:0.02 ug/ml reads 0.20 abs.
Instrument Detection Limit: 0.001 ug/ml
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.01 - 0.6 mg/L
Accuracy- 99% (EPA Standard)
Precision of Controls-

	A	B
mean	.066 mg/L	.167
std. dev.	.005	.008
R.S.D.	7.9 %	5.0 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.009	0.015	0.041
mean	0.083	0.164	0.372

W 0.01 mg/L T 0.05 mg/L

CONTROL LIMITS:

REMARKS:

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

SELENIUM IN SEWAGE

Operating Range = 0.010 to 0.6 mg/L

IN - RUN DUPLICATES

range	<0.010	0.010 to0.12	0.12 to0.30	0.30 to0.6	>0.6
no.	64	31	22	3	2
s.w.		0.0087	0.0145	0.0410	
mean		0.0830	0.1640	0.3720	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
swc1	42	0.066	0.0052	7.88
swc2	43	0.167	0.0084	5.03
475-3	51	0.069	0.0050	7.25

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	0	0.000	0.0000

DATE 87/03/11

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Zinc TEST CODE:ZNUT SAMPLE TYPE:Final Efflnt
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Glass or polyethylene bottle (1 litre)
Preservative- 10 drops of conc. HNO3 in 500 ml of sample.
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.-Yes % Extracted-84.6%
Procedure- Transfer 50 ml of a well mixed sample to a test tube marked at 50 ml and 12.5 ml. Add 1 ml of HNO3 and dry in a forced air oven at 105 °C. Add 2 ml of aqua-regia and digest for 2 hrs.

Cool, and make to final volume of 12.5 ml with distilled water. Allow time for particles to settle and analyze by ICP-emission spectroscopy.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.02 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.001 - 1.00 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	.655 mg/L	
std. dev.	.080 mg/L	
R.S.D.	12.2 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.018	0.047	0.056
mean	0.046	0.353	0.757

W 0.01 mg/L T 0.05 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference (Ref QCFE1) exceeds ±15% RSD.

REMARKS:
- Extraction efficiency measured by spike recovery.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC IN FINAL EFFLUENT

Operating Range = 0.001 to 1.0 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.20	0.20 to0.50	0.50 to1.0	>1.0
no.	5	38	3	3	2
s.w.		0.0179	0.0468	0.0558	
mean		0.0460	0.3530	0.7570	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 31	35	0.655	0.0799	12.20

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	30	0.047	0.0855

DATE 87/01/21

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Total cyanide TEST CODE: CCNAUR SAMPLE TYPE:Sewage
UNIT: QC-Project SUPERVISOR: J. Hipfner

METHOD CODE:001AC2
REVISION NO: DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml
Container- Glass or plastic (preferred)
Preservative- NaOH
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.- % Extracted-100
Procedure- The sample is first run to see if there is cyanide present. The is run directly by the automated high temperature distillation with 25% H_3PO_4-5% H_3PO_2 followed by a colourimetric analysis with chloramine T -isonicotinic acid -barbituric acid method.
If the total cyanide is > .01 mg/L then 5 to 250 ml of sample is manually distilled with 30 ml of 15%(w/v)tartaric acid. The distillate is collected in 50 ml of 1N NaOH, and analyzed by the automated Technicon distillation system referred to above.

INTERFERENCES: SCN interference is removed by distillation. Distillable organics may interfere; also S= at high levels.
REPORTING RESULTS:Mg/l CN: 3 decimal places up to 3 significant figs
INSTRUMENTATION: Technicon AAII continuous flow analyzer including pump, colourimeter, appropriate autosampler and recorder. High temperature distillation apparatus (Technicon). Manual dist. app
Calibration Range: 0 to 0.4 mg/l as CN
Resolution: 0.001 mg/l
Sensitivity:
Instrument Detection Limit: 0.001 mg/l
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.00100 to 0.400 mg/l
Accuracy- 100%
Precision of Controls-

	A	B
mean	.110 mg/L	0.059
std. dev.	.0027mg/L	0.0026
R.S.D.	2.45 %	4.41 %

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0011	0.0009	0.0127
mean	0.014	0.119	0.293

W .001 mg/L T .005 mg/L

CONTROL LIMITS:

REMARKS: Pure CN standards are recovered 100% during manual distillation. Complex cyanides can normally be expected to be recovered at 100%.

SUMMARY REPORT OF QUALITY CONTROL DATA

TOTAL CYANIDE IN SEWAGE

Operating Range = 0.001 to 0.4 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.08	0.08 to0.2	0.2 to0.4	>0.4
no.	0	44	2	0	0
s.w.		0.0009	0.0041	0	
mean		0.0141	0.089	0	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qc-a	146	0.149	0.0049	3.29
qc-b	146	0.018	0.0022	12.22

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	146	0.001	0

DATE 88/06/01

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME: Free cyanide TEST CODE: CCNFUR SAMPLE TYPE: Sewage
UNIT: QC-Project SUPERVISOR: J. Hipfner

METHOD CODE:700AC2
REVISION NO: DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 100 ml
Container- Glass or plastic (preferred)
Preservative- NaOH
Other-

SAMPLE PREPARATION:Partial Extn.- Total Extn.- % Extracted- *
Procedure- Pass sample aliquot through an automated low temperature distillation (106°C) in a distillation acid consisting of 10% acetic acid and 0.5% zinc acetate.
Analyze distillate by the Chloramine-T -pyridine-barbituric acid colourometric method, or equivalent.

INTERFERENCES: None

REPORTING RESULTS:Mg/l CN to 2 decimal places up to 3 significant figs
INSTRUMENTATION: Technicon automated continuous flow analyzer including pump, colourimetric distillation apparatus and sampler; suitable recorder.
Calibration Range: 0 to 0.4 mg/l as CN
Resolution: 0.001
Sensitivity:
Instrument Detection Limit: 0.001 mg/l
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.0100 to 0.400 mg/L
Accuracy- 100%
Precision of Controls-

	A	B
mean	.110 mg/L	0.060
std. dev.	.0036mg/L	0.0031
R.S.D.	3.27 %	5.17%

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.0004	0.0007	0.0000
mean	0.0135	0.098	0.290

W .001 mg/L T .005 mg/L

CONTROL LIMITS:

REMARKS:* The test defines the results reported in this case. The terminology "Weak Acid Dissociable" is commonly used and represents weakly associated cyanide compounds such as KCN, NaCN, NiCN4, HCN,etc.

SUMMARY REPORT OF QUALITY CONTROL DATA

FREE CYANIDE IN SEWAGE

Operating Range = 0.001 to 0.4 mg/L

IN - RUN DUPLICATES

range	<0.001	0.001 to0.08	0.08 to0.2	0.2 to0.4	>0.4
no.	0	44	1	1	0
s.w.		0.0011	0.0141	0.0078	
mean		0.0069	0.13	0.3485	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
qc-a	135	0.151	0.0062	4.11
qc-b	135	0.018	0.0022	12.22

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLK	135	0.001	0

DATE 88/06/01

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Aluminum TEST CODE:ALUT SAMPLE TYPE:Sludge;Cake
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml; 100 g filter cake
Container- Glass or polyethylene bottle (1 litre)
Preservative- None
Other- Fill only 2/3rd of the container for sludge

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- ND
Procedure- Blend 30 g of filter cake with 270 ml distilled water in a Waring blender. Dry 5 ml of diluted filter cake or 5 ml of sludge overnight in a test tube marked at 25 ml using a forced air oven set at 110°C.
Add 1 ml HNO3 and 3 ml HCl and digest the sample for 6 hrs.
Dilute the digest to 25 ml with distilled water and filter through a Whatman #40 filter paper.
Analyze the filtrate by plasma emission spectrometry as part of a multi-element measurement system.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 100 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.5 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.4 - 1000 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	861 mg/L	
std. dev.	70 mg/L	
R.S.D.	8.2 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	20	27	26
mean	104	313	735

W 0.5 mg/L T 2.5 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

ALUMINUM IN SLUDGE

Operating Range = 0.400 to 1000.0 mg/L

IN - RUN DUPLICATES

range	<0.400	0.400 to200.00	200.00to500.00	500.00to1000.0	>1000.0
no.	1	25	26	13	40
s.w.		20.3035	26.5879	25.7917	
mean		104.2690	313.0810	734.8460	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 29	107	860.954	70.4773	8.19

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	139	1.747	1.1485

DATE 87/01/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Cadmium TEST CODE:CDUT SAMPLE TYPE:Sludge;Cake
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml; 100 g filter cake
Container- Glass or polyethylene bottle (1 litre)
Preservative- None
Other- Fill only 2/3rd of the container for sludge

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- ND
Procedure- Blend 30 g of filter cake with 270 ml distilled water in a Waring blender. Dry 5 ml of diluted filter cake or 5 ml of sludge overnight in a test tube marked at 25 ml using a forced air oven set at 110°C.
Add 1 ml HNO_3 and 3 ml HCl and digest the sample for 6 hrs.
Dilute the digest to 25 ml with distilled water and filter through a Whatman #40 filter paper.
Analyze the filtrate by plasma emission spectrometry as part of a multi-element measurement system.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 0.4 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.01 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range-0.025 to 5.0 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	0.70 mg/L	
std. dev.	0.10 mg/L	
R.S.D.	14.3 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.09	0.07	0.14
mean	0.20	1.38	3.76

W 0.02 mg/L T 0.10 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

CADMIUM IN SLUDGE

Operating Range = 0.025 to 5.0 mg/L

IN - RUN DUPLICATES

range	<0.025	0.025 to1.00	1.00 to2.50	2.50 to5.0	>5.0
no.	23	68	3	9	2
s.w.		0.0929	0.0660	0.1431	
mean		0.2020	1.3840	3.7640	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 29	108	0.704	0.1008	14.32

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	5	0.013	0.0021

DATE 87/01/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Cobalt TEST CODE:COUT SAMPLE TYPE:Sludge;Cake
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml; 100 g filter cake
Container- Glass or polyethylene bottle (1 litre)
Preservative- None
Other- Fill only 2/3rd of the container for sludge

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- ND
Procedure- Blend 30 g of filter cake with 270 ml distilled water in a Waring blender. Dry 5 ml of diluted filter cake or 5 ml of sludge overnight in a test tube marked at 25 ml using a forced air oven set at 110°C.
Add 1 ml HNO_3 and 3 ml HCl and digest the sample for 6 hrs.
Dilute the digest to 25 ml with distilled water and filter through a Whatman #40 filter paper.
Analyze the filtrate by plasma emission spectrometry as part of a multi-element measurement system.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 0.4 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.02 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.1 - 20 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	0.30 mg/L	
std. dev.	0.09 mg/L	
R.S.D.	28.6 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.06		
mean	0.38		

W 0.1 mg/L T 0.5 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

COBALT IN SLUDGE

Operating Range = 0.100 to 20.0 mg/L

IN - RUN DUPLICATES

range	<0.100	0.100 to4.00	4.00 to10.00	10.00 to20.0	>20.0
no.	88	17	0	0	0
s.w.		0.0644	0.0000	0.0000	
mean		0.3800	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 29	103	0.300	0.0859	28.63

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	19	0.027	0.0072

DATE 87/01/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Chromium TEST CODE:CRUT SAMPLE TYPE:Sludge;Cake
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml; 100 g filter cake
Container- Glass or polyethylene bottle (1 litre)
Preservative- None
Other- Fill only 2/3rd of the container for sludge

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- ND
Procedure- Blend 30 g of filter cake with 270 ml distilled water in a Waring blender. Dry 5 ml of diluted filter cake or 5 ml of sludge overnight in a test tube marked at 25 ml using a forced air oven set at 110°C.
Add 1 ml HNO_3 and 3 ml HCl and digest the sample for 6 hrs.
Dilute the digest to 25 ml with distilled water and filter through a Whatman #40 filter paper.
Analyze the filtrate by plasma emission spectrometry as part of a multi-element measurement system.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2.0 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.03 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.05 - 100 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	60.6 mg/L	
std. dev.	7.3 mg/L	
R.S.D.	12.1 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.5	4.8	2.8
mean	4.2	29.1	59.5

W 0.2 mg/L T 1.0 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

CHROMIUM IN SLUDGE

Operating Range = 0.050 to 100.0 mg/L

IN - RUN DUPLICATES

range	<0.050	0.050 to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	4	87	8	4	2
s.w.		0.4730	4.7501	2.8406	
mean		4.1600	29.0790	59.4710	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 29	108	60.604	7.3322	12.10

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	6	0.099	0.0869

DATE 87/01/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Copper TEST CODE:CUUT SAMPLE TYPE:Sludge;Cake
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml; 100 g filter cake
Container- Glass or polyethylene bottle (1 litre)
Preservative- None
Other- Fill only 2/3rd of the container for sludge

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- ND
Procedure- Blend 30 g of filter cake with 270 ml distilled water in a Waring blender. Dry 5 ml of diluted filter cake or 5 ml of sludge overnight in a test tube marked at 25 ml using a forced air oven set at 110°C.
Add 1 ml HNO3 and 3 ml HCl and digest the sample for 6 hrs.
Dilute the digest to 25 ml with distilled water and filter through a Whatman #40 filter paper.
Analyze the filtrate by plasma emission spectrometry as part of a multi-element measurement system.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2.0 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.02 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.05 - 100 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	36.3 mg/L	
std. dev.	4.1 mg/L	
R.S.D.	11.1 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	1.3	3.1	3.1
mean	9.7	28.7	75.0

W 0.1 mg/L T 0.5 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.
- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

COPPER IN SLUDGE

Operating Range = 0.050 to 100.0 mg/L

IN - RUN DUPLICATES

range	<0.050	0.050 to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	1	59	33	10	2
s.w.		1.2541	3.1061	3.0653	
mean		9.7070	28.6830	74.9780	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 29	108	36.306	4.0517	11.16

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	3	0.129	0.0919

DATE 87/01/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Iron TEST CODE:FEUT SAMPLE TYPE:Sludge;Cake
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml; 100 g filter cake
Container- Glass or polyethylene bottle (1 litre)
Preservative- None
Other- Fill only 2/3rd of the container for sludge

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- ND
Procedure- Blend 30 g of filter cake with 270 ml distilled water in a Waring blender. Dry 5 ml of diluted filter cake or 5 ml of sludge overnight in a test tube marked at 25 ml using a forced air oven set at 110°C.
Add 1 ml HNO_3 and 3 ml HCl and digest the sample for 6 hrs.
Dilute the digest to 25 ml with distilled water and filter through a Whatman #40 filter paper.
Analyze the filtrate by plasma emission spectrometry as part of a multi-element measurement system.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 100 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.5 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.1 - 5000 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	2831 mg/L	
std. dev.	269 mg/L	
R.S.D.	9.5 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	21	668	396
mean	287	1912	3312

W 0.5 mg/L T 2.5 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

IRON IN SLUDGE

Operating Range = 0.100 to 5000.0 mg/L

IN - RUN DUPLICATES

range	<0.100	0.100 to1000.0	1000.0to2500.0	2500.0to5000.0	>5000.0
no.	2	61	23	12	7
s.w.		21.1119	668.1845	396.2887	
mean		287.4750	1911.870	3312.121	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 29	107	2830.936	268.6222	9.49

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	9	2.370	3.7840

DATE 87/01/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Lead TEST CODE:PBUT SAMPLE TYPE:Sludge;Cake
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml; 100 g filter cake
Container- Glass or polyethylene bottle (1 litre)
Preservative- None
Other- Fill only 2/3rd of the container for sludge

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- ND
Procedure- Blend 30 g of filter cake with 270 ml distilled water in a Waring blender. Dry 5 ml of diluted filter cake or 5 ml of sludge overnight in a test tube marked at 25 ml using a forced air oven set at 110°C.
Add 1 ml HNO3 and 3 ml HCl and digest the sample for 6 hrs.
Dilute the digest to 25 ml with distilled water and filter through a Whatman #40 filter paper.
Analyze the filtrate by plasma emission spectrometry as part of a multi-element measurement system.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2.0 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.15 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.1 - 50 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	39.6 mg/L	
std. dev.	4.3 mg/L	
R.S.D.	10.8 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	1.0	1.2	1.2
mean	3.9	14.6	34.7

W 0.5 mg/L T 2.5 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

LEAD IN SLUDGE

Operating Range = 0.100 to 50.0 mg/L

IN - RUN DUPLICATES

range	<0.100	0.100 to10.00	10.00 to25.00	25.00 to50.0	>50.0
no.	6	78	10	5	6
s.w.		1.0423	1.1933	1.2037	
mean		3.9100	14.6190	34.7090	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 29	108	39.557	4.2765	10.81

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	3	0.201	0.0450

DATE 87/01/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Manganese TEST CODE:MNUT SAMPLE TYPE:Sludge;Cake
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml; 100 g filter cake
Container- Glass or polyethylene bottle (1 litre)
Preservative- None
Other- Fill only 2/3rd of the container for sludge

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- ND
Procedure- Blend 30 g of filter cake with 270 ml distilled water in a Waring blender. Dry 5 ml of diluted filter cake or 5 ml of sludge overnight in a test tube marked at 25 ml using a forced air oven set at 110°C.
Add 1 ml HNO_3 and 3 ml HCl and digest the sample for 6 hrs.
Dilute the digest to 25 ml with distilled water and filter through a Whatman #40 filter paper.
Analyze the filtrate by plasma emission spectrometry as part of a multi-element measurement system.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2.0 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit:0.01 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.05 - 100 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	33.2 mg/L	
std. dev.	2.7 mg/L	
R.S.D.	8.2 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.6	1.2	4.4
mean	7.7	31.4	57.6

W 0.05 mg/L T 0.25 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

MANGANESE IN SLUDGE

Operating Range = 0.050 to 100.0 mg/L

IN - RUN DUPLICATES

range	<0.050	0.050 to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	0	80	17	4	4
s.w.		0.6154	1.1839	4.4102	
mean		7.6690	31.4420	57.6150	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 29	106	33.186	2.7331	8.24

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	4	1.210	1.8138

DATE 87/01/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Molybdenum TEST CODE:MOUT SAMPLE TYPE:Sludge;Cake
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml; 100 g filter cake
Container- Glass or polyethylene bottle (1 litre)
Preservative- None
Other- Fill only 2/3rd of the container for sludge

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- ND
Procedure- Blend 30 g of filter cake with 270 ml distilled water in a Waring blender. Dry 5 ml of diluted filter cake or 5 ml of sludge overnight in a test tube marked at 25 ml using a forced air oven set at 110°C.
Add 1 ml HNO_3 and 3 ml HCl and digest the sample for 6 hrs.
Dilute the digest to 25 ml with distilled water and filter through a Whatman #40 filter paper.
Analyze the filtrate by plasma emission spectrometry as part of a multi-element measurement system.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 0.4 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.05 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range-0.025 to 10.0 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	.635 mg/L	
std. dev.	.139 mg/L	
R.S.D.	21.8 %	

Precision of Duplicates-low range mid range high range

s.d.	0.190		
mean	0.337		
W 0.2 mg/L		T 1.0 mg/L	

CONTROL LIMITS: Analysis repeated if values for in-house reference exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

MOLYBDENUM IN SLUDGE

Operating Range = 0.025 to 10.0 mg/L

IN - RUN DUPLICATES

range	<0.025	0.025 to2.00	2.00 to5.00	5.00 to10.0	>10.0
no.	45	59	0	0	1
s.w.		0.1904	0.0000	0.0000	
mean		0.3370	0.0000	0.0000	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 29	108	0.635	0.1387	21.84

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	4	0.060	0.0063

DATE 87/01/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Nickel TEST CODE:NIUT SAMPLE TYPE:Sludge;Cake
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml; 100 g filter cake
Container- Glass or polyethylene bottle (1 litre)
Preservative- None
Other- Fill only 2/3rd of the container for sludge

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- ND
Procedure- Blend 30 g of filter cake with 270 ml distilled water in a Waring blender. Dry 5 ml of diluted filter cake or 5 ml of sludge overnight in a test tube marked at 25 ml using a forced air oven set at 110°C.
Add 1 ml HNO_3 and 3 ml HCl and digest the sample for 6 hrs.
Dilute the digest to 25 ml with distilled water and filter through a Whatman #40 filter paper.
Analyze the filtrate by plasma emission spectrometry as part of a multi-element measurement system.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2.0 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.06 mg/L
PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.15 - 50.0 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	23.4 mg/L	
std. dev.	2.4 mg/L	
R.S.D.	10.4 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	0.4	0.7	0.7
mean	1.4	14.8	31.4

W 0.1 mg/L T 0.5 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

NICKEL IN SLUDGE

Operating Range = 0.150 to 50.0 mg/L

IN - RUN DUPLICATES

range	<0.150	0.150 to10.00	10.00 to25.00	25.00 to50.0	>50.0
no.	29	69	5	1	1
s.w.		0.4425	0.6858	0.7354	
mean		1.4280	14.8320	31.4300	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 29	108	23.430	2.4461	10.44

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	7	0.101	0.0386

DATE 87/01/22

ANALYTICAL PROCEDURE
Inorganic Trace Contaminants Section

TEST NAME:Zinc TEST CODE:ZNUT SAMPLE TYPE:Sludge;Cake
UNIT:Ind.,Dom.,Landfill Wastes SUPERVISOR:J. Pimenta

METHOD CODE:
REVISION NO: Original DATE:
NATURE OF LAST REVISION:

SAMPLE HANDLING:
Quantity Required- 500 ml; 100 g filter cake
Container- Glass or polyethylene bottle (1 litre)
Preservative- None
Other- Fill only 2/3rd of the container for sludge

SAMPLE PREPARATION:Partial Extn.-Yes Total Extn.- % Extracted- ND
Procedure- Blend 30 g of filter cake with 270 ml distilled water in a Waring blender. Dry 5 ml of diluted filter cake or 5 ml of sludge overnight in a test tube marked at 25 ml using a forced air oven set at 110°C.
Add 1 ml HNO_3 and 3 ml HCl and digest the sample for 6 hrs.
Dilute the digest to 25 ml with distilled water and filter through a Whatman #40 filter paper.
Analyze the filtrate by plasma emission spectrometry as part of a multi-element measurement system.

INTERFERENCES: Several, compensated for by the computer program.

REPORTING RESULTS: mg/L to 2 places after decimal.
INSTRUMENTATION: Inductively coupled plasma emission spectrometer, Atomscan 2400,equipped with autosampler and DEC computer system for concentration print-out; Apple microcomputer interface to LIS.
Calibration Range: 0 - 2.0 mg/L
Resolution:
Sensitivity:
Instrument Detection Limit: 0.02 mg/L

PERFORMANCE CHARACTERISTICS:
Routine Operating Range- 0.025 - 100 mg/L
Accuracy- Not known; no reference standards available
Precision of Controls-

	A	B
mean	95.3 mg/L	
std. dev.	9.6 mg/L	
R.S.D.	10.0 %	

Precision of Duplicates-

	low range	mid range	high range
s.d.	1.0	1.2	3.3
mean	11.2	31.0	63.8

W 0.05 mg/L T 0.25 mg/L

CONTROL LIMITS: Analysis repeated if values for in-house reference exceeds ±15% RSD.

REMARKS:
- Extraction efficiency not known as no reference standards available.

- Detection Limit - 3x std. dev. of low range within-run duplicates.
- Accuracy - Ratio of mean and cert. value in ref. mat. (%).

SUMMARY REPORT OF QUALITY CONTROL DATA

ZINC IN SLUDGE

Operating Range = 0.025 to 100.0 mg/L

IN - RUN DUPLICATES

range	<0.025	0.025 to20.00	20.00 to50.00	50.00 to100.0	>100.0
no.	0	61	29	7	8
s.w.		1.0233	1.1771	3.2906	
mean		11.1880	30.9840	63.7810	

QA CONTROL SAMPLES

SAMPLE I.D.	NO.	MEAN	STD. DEV.	R.S.D.
REF 29	108	95.339	9.5517	10.02

BLANKS

BLANK I.D.	NO.	MEAN	STD. DEV.
BLANK	40	0.059	0.0989

DATE 87/01/22